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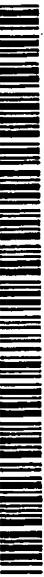
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(54) Title: PRESERVING COMPOSITIONS

(57) Abstract: The specification describes wood preserving compositions comprising basic aqueous solutions of zinc and boron compounds. In particular the specification describes wood preserving compositions comprising zinc borate dissolved in aqueous ammonia. Other preservatives such as copper and quaternary ammonium compounds may also be included. Wood impregnated with the preservative composition is resistant to leaching.

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PRESERVING COMPOSITIONS

The present invention relates to preserving compositions for porous products, especially wood.

5 Compounds of boron have been used in the preservation of wood for a number of years. However, one of the difficulties experienced in use of these compounds is their propensity to leach from the substrate into which they have been impregnated. Consequently, compounds of
10 boron have not been particularly useful in preserving timber exposed to water, for example, pylons embedded in a sea bed.

15 Basic zinc compounds also have toxic efficacy with respect to wood destroying organisms, both in the marine and land environments. However, despite this efficacy having been known, basic zinc compounds have not been used commercially.

20 The present invention is based on the discovery that a composition comprising a basic zinc compound and a compound of boron has a toxic efficacy to wood destroying organisms, both in the marine and land environments with the added benefit of a reduction in the rate at which the boron compound leaches from the impregnated substrate.

25 Accordingly, the present invention provides a preserving composition which includes a basic zinc compound and a compound of boron.

30 A preferred composition comprises zinc ions and borate ions in an aqueous solution of ammonia. The zinc ions may be derived from any soluble zinc compound and the borate ions may be derived from any soluble borate but zinc borate is preferred. Examples of soluble zinc compounds include zinc sulfate and zinc chloride and examples of soluble borates include sodium borate and potassium borate. A solution of zinc ions and borate ions in aqueous ammonia may be prepared by dissolving zinc borate in an aqueous solution of ammonia at a temperature of about 66°C. Alternatively, a soluble zinc salt may be
35

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combined with a soluble borate salt in an aqueous ammonia solution at a temperature of about 66°C.

The preserving composition of the present invention may be combined with other additives having efficacy as preservatives. These include copper sulfate, fluorides such as potassium fluoride and quaternary ammonium compounds.

Porous compounds, especially porous cellulosic compounds, may be impregnated with the preserving composition of the present invention by flooding the porous compound at reduced pressure. Preferably, the pressure is reduced to a level in the range between 0.5 kPa and 0.9 kPa.

In a further aspect, the present invention provides a means of impregnating a substrate with a preserving composition by means of coacervation. Coacervation occurs when a liquid compound separates into two phases below a particular temperature. One phase is rich in one compound and the other is leaner. Coacervation is similar to crystallisation from a mother liquor.

The following examples illustrate the invention.

EXAMPLE 1

25 Preparation of Impregnant

- o 1000 grams of ammonia solution (28%) was added to 1000 grams of water.
- o With constant agitation using a magnetic stirrer, 222.5 grams of zinc borate were added to the diluted ammonia solution.
- o The solution was heated gently at 66°C to form a clear solution.

35 Preparation of Wood Blocks

- o *Pinus radiata* clear sapwood was used for the impregnation, and all the wood blocks were conditioned

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to less than 15% moisture level.

- The numbered blocks prepared for the impregnation were: 028, 107, 147, 282 and 307.
- The volume of the blocks was measured and the results 5 are given below. Although the volume of each block varies slightly, the volume of every individual block was measured to ensure the accuracy of retention calculated.

10

| Block No. | 028 | 107 | 147 | 282 | 307 |
|---------------------------------|--------|--------|--------|--------|--------|
| Volume ($m^3 \times 10^{-6}$) | 12.675 | 12.521 | 12.649 | 12.684 | 12.848 |

- The weight of the blocks was determined

Impregnation

- Once a vacuum of 0.9 kPa was reached, the vacuum pump 15 was kept running for 30 minutes, and the lowest vacuum achieved before flooding was 0.65 kPa.
- Flooding was commenced under vacuum.
- Vacuum was released to atmospheric pressure.
- Atmospheric pressure was employed for 60 minutes.
- The solution was sucked back to the work vessel under 20 vacuum at the end of the pressure period.
- Vacuum was applied to the pressure vessel containing the treated blocks.
- After a vacuum of 2.5 kPa was reached, the vacuum was 25 run for 30 minutes.
- The lowest vacuum reached was 2.1 kPa before the vacuum was released.
- The weight of blocks was determined, and the uptake and 30 retention of chemicals calculated. The results are as follows.

| Block No. | 107 | 307 | 028 | 282 | 147 |
|------------|--------|--------|--------|--------|--------|
| Uptake (g) | 7.7165 | 9.0291 | 8.3166 | 8.084 | 7.8495 |
| Retention | 616.28 | 702.76 | 656.14 | 637.34 | 620.56 |

(kg/m³)

- Blocks were dried naturally in the open air.

Leaching

- The leaching was conducted as detailed in the American
5 Wood Preserver's Association Standard E11-8.
- The blocks were impregnated with distilled water in
exactly the same way as in the impregnation with
preservatives.
- Once a vacuum of 1.5 kPa was reached, the vacuum pump
10 was kept running for 30 minutes, and the lowest vacuum
achieved before flooding was 1.3 kPa.
- Distilled water was used for the flooding.
- Vacuum was released to atmospheric pressure.
- The amount of water used was 50 ml per block, and the
15 total amount of water used was 300 ml. The loss of
water to the uptake was re-added.
- Once the leaching was commenced, the leachant was
changed at intervals of 6 hours, 24 hours and every 48
hours afterwards, and the leachates were collected for
20 analysis.

Analysis of Impregnant, Leachates and Digested Blocks after
Leaching

All the analyses were carried out using
25 techniques of AAS (Atomic Absorption Spectrometry) and ICP-
AES (Inductively Coupled Plasma-Atomic Emission
Spectrometry) with the final results based on ICP analysis.

1. *Analysis of impregnant*

- The impregnant was diluted to an appropriate
concentration that is within the working range of the
analytical instrument.
- The diluted impregnant was determined and the
concentrations of boron and zinc are 12.07g/L and
35 27.89g/L respectively.
- The determined concentration of the impregnant was used

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to calculate the mass balance.

2. *Analysis of leachates*

5 • The leachates were analysed directly without any further sample preparation.

• The concentrations of boron and zinc in the leachates are as follows.

10

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 |
|----------|-----|------|------|------|------|------|------|------|------|
| B (ppm) | 204 | 192 | 202 | 107 | 62.5 | 48.3 | 37.4 | 29.7 | 26.6 |
| Zn (ppm) | 113 | 90.7 | 93.9 | 16.5 | 16.9 | 14.0 | 13.4 | 9.80 | 9.35 |

3. *Analysis of digested wood blocks*

15 • Four out of five blocks were used for the digestion and analysis, and the other one was used for penetration test.

• The four blocks were quantitatively powdered, homogenised and dried at 100°C for 2 hours before analysis.

20 • 3.0000 grams of wood powder were taken for the digestion.

• The digestion was carried out based on the American Wood Preserver's Association Standard A7-93 using nitric acid, sulfuric acid and perchloric acid.

25 • The solutions obtained from the digestion were analysed for boron and zinc and the results are as follows.

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| Block No. | 028 | 107 | 147 | 307 |
|--------------------------------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 197 | 153 | 147 | 219 |
| Total B in each block (mg) | 55.23 | 44.66 | 43.74 | 57.13 |
| Zn in digestion solution (ppm) | 770 | 729 | 744 | 858 |
| Total Zn in each block (mg) | 215.7 | 212.7 | 217.2 | 224.1 |

Summary of Results

| | | |
|----|---------------------------------|-------------------------|
| B | Total boron uptake | 397.2 mg |
| | Total boron in wood | 200.8 mg |
| | Boron mass fraction in wood | 0.479 |
| | Total boron in leachate | 218.1 mg |
| | Boron mass fraction in leachate | 0.521 |
| | Mass balance | 105.5 % |
| | Boron retention after leaching | 3.96 kg/m ³ |
| Zn | Total zinc uptake | 918.0 mg |
| | Total zinc in wood | 869.7 mg |
| | Zinc mass fraction in wood | 0.906 |
| | Total zinc in leachate | 90.61 mg |
| | Zinc mass fraction in leachate | 0.094 |
| | Mass balance | 104.6% |
| | Zinc retention after leaching | 17.16 kg/m ³ |

5

Comment

Overloading could have been committed resulting in a relatively high mass fraction of boron being leached out. The impregnation procedure followed the Bethell full-cell schedule. The severe overloading occurred and some boron leached as a consequence of excess preservative compound being present within the vessels.

The boron retention after leaching, however, is still very high, 3.66 kg/m³, compared with 1 kg/m³ required

by an American company.

EXAMPLE 2

5 Preparation of Impregnant

The impregnant used in Example 2 was prepared in the same way as in Example 1.

- 1000 grams of ammonia solution (28%) was added to 1000 grams of water.
- 10 • With constant agitation using a magnetic stirrer, 222.5 grams of zinc borate were added to the diluted ammonia solution.
- The solution was heated gently at 66°C to form a clear solution.

15

Preparation of Wood Blocks

- Very similar procedure to that in Example 1 was used to prepare the blocks.
- The numbered blocks prepared for the impregnation were: 20 062, 100, 206, 223 and 239.
- The volume and the moisture of the blocks were measured.
- The weight of the blocks was determined.

Impregnation

25 While the impregnation was conducted in a similar way to that in Example 1, different vacuums were used.

- Once a vacuum of 2.1 kPa was reached, the vacuum pump was kept running for 30 minutes, and the lowest vacuum achieved before flooding was 1.9 kPa.
- 30 • Flooding was commenced under vacuum.
- Vacuum was released to atmospheric pressure after flooding completed.
- Atmospheric pressure was employed for 60 minutes.
- The solution was sucked back to the work vessel under vacuum at the end of the pressure period.
- 35 • Vacuum was applied to the pressure vessel containing the treated blocks.

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- After a vacuum of 2.5 kPa was reached, the vacuum was run for 30 minutes.
- The lowest vacuum reached was 2.3 kPa before the vacuum was released.

5 ◦ The weight of blocks was determined, and the uptake and retention of chemicals calculated. The results are as follows.

| Block No. | 062 | 100 | 206 | 223 | 239 |
|-----------------------------------|--------|--------|--------|--------|--------|
| Uptake (g) | 8.8483 | 5.4833 | 8.0169 | 6.9345 | 8.2680 |
| Retention (kg/m ³) | 702.86 | 433.94 | 629.96 | 553.03 | 663.35 |

10 ◦ Blocks were dried naturally in the open air.

Leaching

The leaching was carried out in a similar way to that in Example 1 except different vacuums were used.

15 ◦ The leaching was conducted as detailed in the American Wood Preserver's Association Standard E11-8.

◦ The blocks were impregnated with distilled water in exactly the same way as in the impregnation with preservatives.

20 ◦ Once a vacuum of 1.6 kPa was reached, the vacuum pump was kept running for 30 minutes, and the lowest vacuum achieved before flooding was 1.4 kPa.

◦ Distilled water was used for the flooding.

◦ Vacuum was released to atmospheric pressure.

25 ◦ The amount of water used was 50 ml per block, and the total amount of water used was 300 ml. The loss of water to the uptake was re-added.

◦ Once the leaching was commenced, the leachant was changed at intervals of 6 hours, 24 hours and every 48 hours afterwards, and the leachates were collected for analysis.

Analysis of Impregnant, Leachates and Digested Blocks after Leaching

5 All the analyses were carried out using techniques of AAS (Atomic Absorption Spectrometry) and ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry) with the final results based on ICP analysis.

1. *Analysis of impregnant*

10 • The impregnant was diluted to an appropriate concentration that is within the working range of the analytical instrument.

• The diluted impregnant was determined and the concentrations of boron and zinc are 11.30g/L and 28.28g/L respectively.

15 • The determined concentration of the impregnant was used to calculate the mass balance.

2. *Analysis of leachates*

20 • The leachates were analysed directly without any further sample preparation.

• The boron concentrations in the leachates are as follows.

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 |
|---------|-----|-----|------|------|------|------|------|------|------|
| B (ppm) | 236 | 170 | 68.1 | 40.4 | 29.4 | 25.3 | 21.9 | 20.6 | 17.3 |

25 3. *Analysis of digested wood blocks*

• Four out of five blocks were used for the digestion and analysis, and the other one was used for penetration test.

• The four blocks were quantitatively powdered, homogenised and dried at 100°C for 2 hours before analysis.

30 • 3.0000 grams of wood powder were taken for the digestion.

• The digestion was carried out based on the American Wood Preserver's Association Standard A7-93 using nitric

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acid, sulfuric acid and perchloric acid.

- The solutions obtained from the digestion were analysed for boron and zinc and the results are as follows.

| Block No. | 100 | 206 | 223 | 239 |
|--------------------------------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 85.3 | 166 | 118 | 165 |
| Total B in each block (mg) | 24.14 | 47.42 | 34.16 | 47.31 |
| Zn in digestion solution (ppm) | 556 | 724 | 672 | 741 |
| Total Zn in each block (mg) | 157.5 | 206.3 | 193.9 | 212.9 |

5

Summary of Results

| | | |
|----|---------------------------------|-------------------------|
| B | Total boron uptake | 324.2 mg |
| | Total boron in wood | 153.0 mg |
| | Boron mass fraction in wood | 0.504 |
| | Total boron in leachate | 150.9 mg |
| | Boron mass fraction in leachate | 0.496 |
| | Mass balance | 93.74 % |
| | Boron retention after leaching | 3.04 kg/m ³ |
| Zn | Total zinc uptake | 811.7 mg |
| | Total zinc in wood | 770.6 mg |
| | Zinc mass fraction in wood | 0.949 |
| | Total zinc in leachate | |
| | Zinc mass fraction in leachate | |
| | Mass balance | |
| | Zinc retention after leaching | 15.30 kg/m ³ |

Comment

10

Overloading could have been committed resulting in a relatively high mass fraction of boron being leached out. The impregnation procedure followed the Bethell full-cell schedule. The severe overloading occurred and some boron leached as a consequence of excess preservative

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compound being present within the vessels.

The boron retention after leaching, however, is still very high, 3.04 kg/m³, compared with 1 kg/m³ required by an American company.

5

EXAMPLE 3

Preparation of Impregnant

10 While the formulation used in this example is different from that used in Examples 1 and 2, the preparation of the impregnant was commenced in the similar way to that in Examples 1 and 2.

- 1000 grams of ammonia solution (28%) was added to 1000 grams of water.
- 15 • With constant agitation using a magnetic stirrer, 222.5 grams of zinc borate were added to the diluted ammonia solution.
- The solution was heated gently at 66°C to form a clear solution.
- 20 • 200 grams of copper sulfate were added gradually and then the solution was cooled to room temperature.

Preparation of Wood Blocks

Same procedures were used to prepare blocks as that used in Examples 1 and 2.

- 25 • The numbered blocks prepared for the impregnation were: 043, 044, 190, 211 and 245.
- The volume and the moisture of the blocks were measured.
- The weight of the blocks was determined.

30

Impregnation

While the impregnation was conducted in a similar way to that in Examples 1 and 2, different vacuums were used.

- 35 • Once a vacuum of 1.9 kPa was reached, the vacuum was maintained for 30 minutes, and the vacuum before flooding was also 1.7 kPa.
- Flooding was commenced under vacuum.

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- Vacuum was released to atmospheric pressure after flooding completed.
- Atmospheric pressure was employed for 60 minutes.
- The solution was sucked back to the work vessel under 5 vacuum at the end of the pressure period.
- Vacuum was applied to the pressure vessel containing the treated blocks.
- After a vacuum of 2.4 kPa was reached, the vacuum was run for 30 minutes.
- 10 ◦ The lowest vacuum reached was 2.3 kPa before the vacuum was released.
- The mass of blocks was determined straight after the pressure treatment, and the uptake and retention of chemicals calculated. The results are as follows.

15

| Block No. | 043 | 044 | 190 | 211 | 245 |
|-----------------------------------|--------|--------|--------|--------|--------|
| Uptake (g) | 7.8235 | 8.2008 | 8.3631 | 7.2498 | 7.3124 |
| Retention (kg/m ³) | 617.58 | 638.19 | 659.65 | 576.98 | 579.43 |

- Blocks were dried naturally in the open air and conditioned for two weeks.

NOTE: The vacuum utilised in this modified Bethell schedule is very important and we have discovered this to be a 20 significant part of this process, ultimately aimed at reduced boron leaching.

Leaching

The leaching was carried out in a similar way to that in 25 Examples 1 and 2 although different vacuums were used.

- The leaching was conducted as detailed in the American Wood Preserver's Association Standard E11-8.
- The blocks were impregnated with distilled water in exactly the same way as in the impregnation with preservatives.
- 30 ◦ Once a vacuum of 1.5 kPa was reached, the vacuum pump was kept running for 30 minutes.

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- Distilled water was used for the flooding.
- Vacuum was released to atmospheric pressure.
- The amount of water used was 50 ml per block, and the total amount of water used was 300 ml. The loss of water to the uptake was re-added.
- Once the leaching was commenced, the leachant was changed at intervals of 6 hours, 24 hours and every 48 hours afterwards, and the leachates were collected for analysis.

10

Analysis of Impregnant, Leachates and Digested Blocks after Leaching

All the analyses were carried out using techniques of AAS (Atomic Absorption Spectrometry) and ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry) with the final results based on ICP analysis.

1. *Analysis of impregnant*
 - The impregnant was diluted to an appropriate concentration that is within the working range of the analytical instrument.
 - The diluted impregnant was determined and the concentrations of boron, zinc and copper were 10.80g/L, 26.94g/L and 36.52g/L respectively.
 - The determined concentration of the impregnant was used to calculate the mass balance.

2. *Analysis of leachates*
 - The leachates were analysed directly without any further sample preparation.
 - The concentrations of boron and copper in the leachates were as follows.

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 | L10 |
|----------|------|------|------|------|------|------|------|------|------|------|
| B (ppm) | 377 | 221 | 92.0 | 53.0 | 37.3 | 5.37 | 13.1 | 9.66 | 8.50 | 5.25 |
| Cu (ppm) | 32.9 | 14.2 | 6.61 | 5.11 | 5.17 | 1.66 | 2.26 | 1.86 | 1.60 | 1.97 |

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3. *Analysis of digested wood blocks*

- Four out of five blocks were used for the digestion and analysis, and the other one was used for a penetration test.
- 5 • The four blocks were quantitatively powdered, homogenised and dried at 100°C for 2 hours before analysis.
- 3.0000 grams of wood powder were taken for the digestion.
- 10 • The digestion was carried out based on the American Wood Preserver's Association Standard A7-93 using nitric acid, sulfuric acid and perchloric acid.
- The solutions obtained from the digestion were analysed for boron, zinc and copper and the results are as
- 15 follows.

| Block No. | 043 | 044 | 190 | 211 |
|--------------------------------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 63.9 | 52.5 | 50.2 | 43.7 |
| Total B in each block (mg) | 18.22 | 16.89 | 16.00 | 14.28 |
| Zn in digestion solution (ppm) | 475 | 542 | 540 | 414 |
| Total Zn in each block (mg) | 135.4 | 174.3 | 172.2 | 135.3 |
| Cu in digestion solution (ppm) | ? | 1092 | 1144 | 920 |
| Total Cu in each block (mg) | 108.6 | 351.3 | 364.9 | 300.5 |

Summary of Results

| | | |
|----|----------------------------------|-------------------------|
| B | Total boron uptake | 341.7 mg |
| | Total boron in wood | 65.38 mg |
| | Boron mass fraction in wood | 0.249 |
| | Total boron in leachate | 197.2 mg |
| | Boron mass fraction in leachate | 0.751 |
| | Mass balance | 76.84 % |
| | Boron retention after leaching | 1.29 kg/m ³ |
| Zn | Total zinc uptake | 852.4 mg |
| | Total zinc in wood | 617.2 mg |
| | Zinc mass fraction in wood | 0.724 |
| | Total zinc in leachate | ? |
| | Zinc mass fraction in leachate | ? |
| | Mass balance | ? |
| | Zinc retention after leaching | 12.16 kg/m ³ |
| Cu | Total copper uptake | 1155 mg |
| | Total copper in wood | 1125 mg |
| | Copper mass fraction in wood | 0.985 |
| | Total copper in leachate | 17.61 mg |
| | Copper mass fraction in leachate | 0.015 |
| | Mass balance | 98.93 % |
| | Copper retention after leaching | 22.17 kg/m ³ |

EXAMPLE 4

5

Preparation of Impregnant

While the formulation used in this example is different from that used in Examples 1, 2 and 3, the impregnant was first prepared as that in Example 3 and then 8.9 grams of 10 potassium fluoride were added.

- 1000 grams of ammonia solution (28%) was added to 1000 grams of water.
- With constant agitation using a magnetic stirrer, 222.5 grams of zinc borate were added to the diluted ammonia 15 solution.
- The solution was heated gently at 66°C to form a clear

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solution.

- 200 grams of copper sulfate were added gradually and then the solution was cooled to room temperature.
- 8.9 grams of potassium fluoride were added.

5

Preparation of Wood Blocks

The procedure used to prepare blocks is very similar to that in Examples 1 and 2.

- The numbered blocks prepared for the impregnation were: 10 066, 143, 200, 205 and 261.
- The volume and the moisture of the blocks were measured.
- The weight of the blocks was determined.

Impregnation

15 While the impregnation was conducted in a similar way to that in Examples 1 and 2, different vacuums were used.

- Once a vacuum of 1.9 kPa was reached, the vacuum pump was kept running for 30 minutes, and the vacuum before flooding was also 1.9 kPa.
- Flooding was commenced under vacuum.
- Vacuum was released to atmospheric pressure after flooding was completed.
- Atmospheric pressure was employed for 60 minutes.
- The solution was sucked back to the work vessel under 25 vacuum at the end of the pressure period.
- Vacuum was applied to the pressure vessel containing the treated blocks.
- After a vacuum of 2.5 kPa was reached, the vacuum was run for 30 minutes.
- The lowest vacuum reached was 2.3 kPa before the vacuum was released.
- The weight of blocks was determined straight after the pressure treatment, and the uptake and retention of chemicals calculated. The results are as follows.

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| Block No. | 066 | 143 | 200 | 205 | 261 |
|-----------------------------------|--------|--------|--------|--------|--------|
| Uptake (g) | 7.2259 | 7.4066 | 7.2929 | 7.1257 | 7.6352 |
| Retention (kg/m ³) | 573.03 | 593.29 | 580.64 | 567.38 | 612.88 |

- Blocks were dried naturally in the open air.

Leaching

The leaching was carried out in a similar way to that used in Example 1 although different vacuums were used.

5 • The leaching was conducted as detailed in the American Wood Preserver's Association Standard E11-8.

• The blocks were impregnated with distilled water in exactly the same way as in the impregnation with preservatives.

10 • Once a vacuum of 0.5 kPa was reached, the vacuum pump was kept running for 30 minutes.

• Distilled water was used for the flooding.

• Vacuum was released to atmospheric pressure.

15 • The amount of water used was 50 ml per block, and the total amount of water used was 300 ml. The loss of water to the uptake was re-added.

• Once the leaching was commenced, the leachant was changed at intervals of 6 hours, 24 hours and every 48 hours afterwards, and the leachates were collected for analysis.

20

Analysis of Impregnant, Leachates and Digested Blocks after Leaching

25 All the analyses were carried out using techniques of AAS (Atomic Absorption Spectrometry) and ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry) with the final results based on ICP analysis.

30 1. *Analysis of impregnant*

- The impregnant was diluted to an appropriate concentration that is within the working range of the

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analytical instrument.

- The diluted impregnant was determined and the concentrations of boron, zinc and copper were 10.63g/L, 27.11g/L and 36.55g/L respectively.
- 5 • The determined concentration of the impregnant was used to calculate the mass balance.

2. *Analysis of leachates*

- The leachates were analysed directly without any further 10 sample preparation.
- The concentrations of boron and copper in the leachates were as follows.

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 |
|----------|------|------|------|------|------|------|------|------|------|
| B (ppm) | 469 | 501 | 48.4 | 20.0 | 20.7 | 13.7 | 7.45 | 5.82 | 6.13 |
| Cu (ppm) | 57.0 | 46.2 | 4.32 | 2.78 | 3.61 | 2.87 | 1.99 | 2.20 | 1.69 |

15 3. *Analysis of digested wood blocks*

- Four out of five blocks were used for the digestion and analysis, and the other one was used for a penetration test.
- The four blocks were quantitatively powdered, 20 homogenised and dried at 100°C for 2 hours before analysis.
- 3.0000 grams of wood powder were taken for the digestion.
- The digestion was carried out based on the American Wood 25 Preserver's Association Standard A7-93 using nitric acid, sulfuric acid and perchloric acid.
- The solutions obtained from the digestion were analysed for boron, zinc and copper and the results are as follows.

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| Block No. | 143 | 200 | 205 | 261 |
|--------------------------------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 61.0 | 40.0 | 47.6 | 53.4 |
| Total B in each block (mg) | 19.39 | 13.60 | 15.51 | 17.59 |
| Zn in digestion solution (ppm) | 410 | 333 | 370 | 467 |
| Total Zn in each block (mg) | 130.2 | 113.3 | 120.6 | 153.8 |
| Cu in digestion solution (ppm) | 45.0 | 29.7 | 646 | 1048 |
| Total Cu in each block (mg) | 134.0 | 86.80 | 252.7 | 345.3 |

Summary of Results

| | | |
|----|----------------------------------|-------------------------|
| B | Total boron uptake | 313.3 mg |
| | Total boron in wood | 66.09 mg |
| | Boron mass fraction in wood | 0.201 |
| | Total boron in leachate | 262.1 mg |
| | Boron mass fraction in leachate | 0.799 |
| | Mass balance | 104.7 % |
| | Boron retention after leaching | 1.32 kg/m ³ |
| Zn | Total zinc uptake | 798.8 mg |
| | Total zinc in wood | 517.9 mg |
| | Zinc mass fraction in wood | 0.648 |
| | Total zinc in leachate | ? |
| | Zinc mass fraction in leachate | ? |
| | Mass balance | ? |
| | Zinc retention after leaching | 10.35 kg/m ³ |
| Cu | Total copper uptake | 1077 mg |
| | Total copper in wood | 818.7 mg |
| | Copper mass fraction in wood | 0.963 |
| | Total copper in leachate | 31.12 mg |
| | Copper mass fraction in leachate | 0.037 |
| | Mass balance | 78.92 % |
| | Copper retention after leaching | 16.35 kg/m ³ |

EXAMPLE 5Preparation of Impregnant

The formulation used in this example is the same as that in
5 Example 3.

- o 1000 grams of ammonia solution (28%) was added to 1000 grams of water.
- o With constant agitation using a magnetic stirrer, 222.5 grams of zinc borate were added to the diluted ammonia
- 10 solution.
- o The solution was heated gently at 66°C to form a clear solution.
- o 200 grams of copper sulfate were added gradually and then the solution was cooled to room temperature.

15

Preparation of Wood Blocks

The procedure used to prepare blocks is very similar to that in the other examples. Ten blocks were used in this example rather than five blocks as in the previous
20 examples.

- o The numbered blocks prepared for the impregnation were: 168, 039, 212, 125, 313, 080, 238, 299, 195 and 051.
- o The volume and the moisture of the blocks were measured.
- o The weight of the blocks was determined.

25

Impregnation

While the impregnation was conducted in a similar way to that used in the other examples, different vacuums were used.

- 30 o Once a vacuum of 1.0 kPa was reached, the vacuum pump was kept running for 30 minutes, and the vacuum before flooding was 0.6 kPa.
- o Flooding was commenced under vacuum.
- o Vacuum was released to atmospheric pressure after flooding completed.
- 35 o Atmospheric pressure was employed for 90 minutes.
- o The solution was sucked back to the work vessel under

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vacuum at the end of the pressure period.

- Vacuum was applied to the pressure vessel containing the treated blocks.
- After a vacuum of 1.3 kPa was reached, the vacuum was run for 30 minutes.
- The lowest vacuum reached was 1.2 kPa before the vacuum was released.
- The weight of blocks was determined straight after the pressure treatment, and the uptake and retention of chemicals calculated. The results are as follows.

| Block No. | 168 | 039 | 212 | 125 | 313 |
|--------------------------------|--------|--------|--------|--------|--------|
| Uptake (g) | 7.8782 | 8.6661 | 8.1115 | 9.0619 | 7.8899 |
| Retention (kg/m ³) | 631.92 | 701.65 | 644.38 | 720.57 | 622.53 |
| Block No. | 080 | 238 | 299 | 195 | 051 |
| Uptake (g) | 7.9759 | 9.4277 | 8.8812 | 9.3488 | 8.9464 |
| Retention (kg/m ³) | 639.15 | 739.31 | 696.62 | 738.04 | 695.30 |

- Blocks were dried naturally in the open air.

Leaching

- 15 The leaching was carried out in a similar way to that in the other examples although different vacuums were used.
 - The leaching was conducted as detailed in the American Wood Preserver's Association Standard E11-8.
 - The blocks were impregnated with distilled water in exactly the same way as in the impregnation with preservatives.
 - Once a vacuum of 0.9 kPa was reached, the vacuum pump was kept running for 30 minutes. A vacuum of 0.6 kPa was achieved before flooding.
- 20 25 • Distilled water was used for the flooding.
 - Vacuum was released to atmospheric pressure.
 - The amount of water used was 50 ml per block, and the

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total amount of water used was 500 ml. The loss of water to the uptake was re-added.

5 ◦ Once the leaching was commenced, the leachant was changed at intervals of 6 hours, 24 hours and every 48 hours afterwards, and the leachates were collected for analysis.

Analysis of Impregnant, Leachates and Digested Blocks after Leaching

10 All the analyses were carried out using techniques of AAS (Atomic Absorption Spectrometry) and ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry) with the final results based on ICP analysis.

15 1. *Analysis of impregnant*

◦ The impregnant was diluted to an appropriate concentration that is within the working range of the analytical instrument.

◦ The diluted impregnant was determined and the concentrations of boron, zinc and copper are 5.61g/L, 21.25g/L and 48.07g/L respectively.

◦ The determined concentration of the impregnant was used to calculate the mass balance.

25 2. *Analysis of leachates*

◦ The leachates were analysed directly without any further sample preparation.

◦ The concentrations of boron, zinc and copper in the leachates are as follows.

30

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 | L10 | L11 |
|----------|------|------|------|------|------|------|------|------|------|------|------|
| B (ppm) | 174 | 186 | 157 | 80.1 | 48.5 | 32.0 | 22.4 | 15.6 | 11.9 | 9.62 | 7.59 |
| Zn (ppm) | 86.5 | 47.4 | 28.2 | 10.9 | 4.07 | 4.49 | 1.26 | 1.38 | 1.04 | 1.51 | 1.14 |
| Cu (ppm) | 131 | 113 | 110 | 59.3 | 26.8 | 18.9 | 5.14 | 2.88 | 1.75 | 2.17 | 1.52 |

3. *Analysis of digested wood blocks*

◦ After the penetration test, all the ten blocks were

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quantitatively powdered, homogenised and dried at 100°C for 2 hours before analysis.

- 3.0000 grams of wood powder were taken for the digestion.

5 • The digestion was carried out based on the American Wood Preserver's Association Standard A7-93 using nitric acid, sulfuric acid and perchloric acid.

• The solutions obtained from the digestion were analysed for boron, zinc and copper and the results are as

10 follows.

| Block No. | 168 | 039 | 212 | 125 | 313 |
|--------------------------------|-------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 38.9 | 40.5 | 39.7 | 50.0 | 35.1 |
| Total B in each block (mg) | 9.733 | 9.866 | 9.387 | 11.91 | 8.326 |
| Zn in digestion solution (ppm) | 539 | 593 | 595 | 632 | 579 |
| Total Zn in each block (mg) | 134.8 | 144.5 | 140.6 | 150.5 | 137.1 |
| Cu in digestion solution (ppm) | 1644 | 1813 | 1898 | 1997 | 1794 |
| Total Cu in each block (mg) | 411.3 | 441.5 | 448.3 | 475.8 | 425.0 |
| Block No. | 080 | 238 | 299 | 195 | 051 |
| B in digestion solution (ppm) | 40.5 | 43.4 | 41.4 | 52.5 | 49.0 |
| Total B in each block (mg) | 9.402 | 10.68 | 10.62 | 12.80 | 12.26 |
| Zn in digestion solution (ppm) | 588 | 626 | 590 | 635 | 611 |
| Total Zn in each block (mg) | 136.5 | 154.1 | 151.5 | 154.8 | 152.6 |
| Cu in digestion solution (ppm) | 1728 | 1807 | 1647 | 1770 | 1757 |
| Total Cu in each block (mg) | 401.1 | 444.5 | 422.6 | 431.7 | 439.2 |

Summary of Results

| | | |
|----|----------------------------------|-------------------------|
| B | Total boron uptake | 483.3 mg |
| | Total boron in wood | 105.0 mg |
| | Boron mass fraction in wood | 0.220 |
| | Total boron in leachate | 372.2 mg |
| | Boron mass fraction in leachate | 0.780 |
| | Mass balance | 98.75 % |
| | Boron retention after leaching | 0.832 kg/m ³ |
| Zn | Total zinc uptake | 1831 mg |
| | Total zinc in wood | 1457 mg |
| | Zinc mass fraction in wood | 0.939 |
| | Total zinc in leachate | 93.97 mg |
| | Zinc mass fraction in leachate | 0.061 |
| | Mass balance | 84.68 % |
| | Zinc retention after leaching | 11.55 kg/m ³ |
| Cu | Total copper uptake | 4143 mg |
| | Total copper in wood | 4341 mg |
| | Copper mass fraction in wood | 0.948 |
| | Total copper in leachate | 236.5 mg |
| | Copper mass fraction in leachate | 0.052 |
| | Mass balance | 110.5 % |
| | Copper retention after leaching | 34.41 kg/m ³ |

Comment

5 • The formulation prepared and utilised as detailed in Examples 3, 4 and 5 all contain copper. Each formulation detailed in every example to date contains zinc. Of great importance is the fact that basic (alkaline) zinc containing compounds have been found to

10 exhibit a high degree of toxic efficacy to wood destroying organisms both in the marine and land environments. We have discovered a way of combining the efficacy of boron within a complex basic polyborate-zinc system which reduces the leaching of boron from

15 commodities treated with this preservative composition. The efficacy of basic zinc containing compositions was

never researched beyond its initial discovery. We have discovered a successful way of complementing the efficacy of boron with that of basic zinc compounds with the unexpected, unique reduction of the leaching of 5 boron, resulting from the interaction of the preservative composition and its impregnated, soaked, brushed, sprayed or diffusion treated substrate.

- Fluorine is an important component of Example 4 in which it contributes additional synergistic efficacy.
- 10 • The formulation utilised in Example 5 was prepared with a lower boron concentration. This constitutes one of several preservative compositions purposely formulated to result in a lower boron retention in timber and other treated commodities which are to be used above ground 15 and especially in building, walkway, bush tracks and numerous other constructions. Especially useful in indoor environments, such compositions will constitute economic protection without any less or reduction in efficacy.

20

EXAMPLE 6

Preparation of Impregnant

The preservative composition was first prepared 25 as detailed in Example 1. 1551 grams of this solution were slowly combined with 30 grams of N-alkyl tallow N dimethyl amino propyl N trimethyl ammonium dichloride in isopropanol water solution. This composition was agitated for several hours until a clear solution was produced. The solution 30 was made up to 2000 grams, by addition of double distilled water. The pH of this composition was determined as 12.6.

Quaternary ammonium salts have long been known to exhibit high surface activity coupled with biological and fungicidal efficacy. Such complex compositions have 35 usually been used to formulate emulsions of cationic nature at low pH. These unique properties have been retained in this complex wood preservative composition. Penetration

and distribution in treated timber have been enhanced and complemented by additional efficacy and boron fixation. However, boron diffusion and movement within the treated commodity has not been hindered, nor fixation rendered so efficient as to hinder efficacy. More surprising and wholly unexpected is the enhanced biocidal efficacy and composition stability in an alkaline environment.

A further very important development, relevant to this composition and previous surface activated compositions such as detailed in Example 7, is their inherent property of self-emulsification with a wide variety of oils which include, but are not limited to, petroleum derived oils, mineral oils, vegetable oils and more especially creosote oils, the latter having great wood preservation significance and limited potential for diversity of formulation and application.

Preparation of Wood Blocks

The procedure used to prepare blocks is very similar to that in the other examples. Totally ten blocks were used in this example.

- o The numbered blocks prepared for the impregnation were: 218, 144, 250, 217, 258, 001, 132, 303, 274 and 226.
- o The volume and the moisture of the blocks were measured.
- o The weight of the blocks was determined.

Impregnation

While the impregnation was conducted in a similar way to that used in the other examples, different vacuums were used.

- o Once a vacuum of 0.9 kPa was reached, the vacuum pump was kept running for 30 minutes, and the vacuum before flooding was also 0.9 kPa.
- o Flooding was commenced under vacuum.
- o Vacuum was released to atmospheric pressure after flooding completed.
- o Atmospheric pressure was employed for 90 minutes.

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- The solution was sucked back to the work vessel under vacuum at the end of the pressure period.
- Vacuum was applied to the pressure vessel containing the treated blocks for 30 minutes, and a vacuum of 1.9 kPa was reached before the vacuum was released.
- The weight of blocks was determined straight after the pressure treatment, and the uptake and retention of chemicals calculated. The results are as follows.

| Block No. | 218 | 144 | 250 | 217 | 258 |
|--------------------------------|--------|--------|--------|---------|---------|
| Uptake (g) | 9.6596 | 9.4843 | 9.6391 | 10.1950 | 8.7983 |
| Retention (kg/m ³) | 757.97 | 755.66 | 758.86 | 799.99 | 699.50 |
| Block No. | 001 | 132 | 303 | 274 | 226 |
| Uptake (g) | 9.6670 | 9.9522 | 9.2082 | 9.6967 | 10.0360 |
| Retention (kg/m ³) | 774.17 | 785.43 | 725.00 | 764.24 | 785.34 |

- Blocks were dried naturally in the open air.

Leaching

All blocks were dried at 108°C for 24 hours before leaching, and the leaching was carried out in a similar way to that in the other examples although different vacuums were used.

- The leaching was conducted as detailed in the American Wood Preserver's Association Standard E11-8.
- The blocks were impregnated with distilled water in the exactly same way as in the impregnation with preservatives.
- Once a vacuum of 1.5 mmHg was reached, the vacuum pump was kept running for 35 minutes. The final vacuum achieved before flooding was also 1.5 mmHg.
- The flooding was conducted under vacuum, and distilled water was used for the flooding.
- Vacuum was released to atmospheric pressure after the

flooding was completed.

- The amount of water used was 50 ml per block, and the total amount of water used was 500 ml. The loss of water to the uptake was re-added.
- 5 ◦ Once the leaching was commenced, the leachant was changed at the intervals of 6 hours, 24 hours and every 48 hours afterwards, and the leachates were collected for analysis.

10 Analysis of Impregnant, Leachates and Digested Blocks after Leaching

15 All the analyses were carried out using techniques of AAS (Atomic Absorption Spectrometry) and ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry) with the final results based on ICP analysis.

1. *Analysis of impregnant*

- The impregnant was diluted to an appropriate concentration that is within the working range of the 20 analytical instrument.
- The diluted impregnant was determined and the concentrations of boron, zinc and copper are 4.43g/L, 17.07g/L and 42.32g/L respectively.
- The determined concentration of the impregnant was used 25 to calculate the mass balance.

2. *Analysis of leachates*

- The leachates were analysed directly without any further sample preparation.
- 30 ◦ The concentrations of boron, zinc and copper in the leachates are as follows.

| Sample | L1 | L2 | L3 | L4 | L5 | L6 | L7 | L8 | L9 |
|----------|------|------|------|------|------|------|------|------|------|
| B (ppm) | 208 | 195 | 134 | 80.3 | 42.0 | 27.3 | 20.5 | 16.2 | 11.4 |
| Zn (ppm) | 85.3 | 62.1 | 38.9 | 18.2 | 7.09 | 5.10 | 2.51 | 2.47 | 2.43 |
| Cu (ppm) | 97.9 | 110 | 85.9 | 50.6 | 19.5 | 11.1 | 1.91 | 1.43 | 1.58 |

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3. *Analysis of digested wood blocks*

- After the penetration test, all the ten blocks were quantitatively powdered, homogenised and dried at 100°C for 2 hours before analysis.
- 5 • 3.0000 grams of wood powder were taken for the digestion.
- The digestion was carried out based on the American Wood Preserver's Association Standard A7-93 using nitric acid, sulfuric acid and perchloric acid.
- 10 • The solutions obtained from the digestion were analysed for boron, zinc and copper and the results are as follows.

| Block No. | 218 | 144 | 250 | 217 | 258 |
|--------------------------------|-------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 31.4 | 19.5 | 23.0 | 29.3 | 15.9 |
| Total B in each block (mg) | 7.487 | 4.789 | 5.683 | 6.942 | 4.266 |
| Zn in digestion solution (ppm) | 623 | 584 | 593 | 636 | 488 |
| Total Zn in each block (mg) | 149.7 | 143.8 | 146.2 | 156.3 | 131.1 |
| Cu in digestion solution (ppm) | 1647 | 1726 | 1692 | 1740 | 1465 |
| Total Cu in each block (mg) | 392.1 | 424.8 | 417.6 | 427.4 | 393.6 |

| Block No. | 001 | 132 | 303 | 274 | 226 |
|--------------------------------|-------|-------|-------|-------|-------|
| B in digestion solution (ppm) | 25.5 | 26.2 | 19.0 | 22.7 | 24.1 |
| Total B in each block (mg) | 6.059 | 6.588 | 5.121 | 5.799 | 6.074 |
| Zn in digestion solution (ppm) | 607 | 601 | 535 | 593 | 625 |
| Total Zn in each block (mg) | 144.5 | 151.4 | 144.2 | 151.7 | 157.4 |

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| | | | | | |
|-----------------------------------|-------|-------|-------|-------|-------|
| Cu in digestion solution (ppm) | 1664 | 1598 | 1455 | 1503 | 1606 |
| Total Cu in each block (mg) | 395.9 | 402.3 | 392.2 | 384.6 | 404.4 |

Summary of Results

| | | |
|----|----------------------------------|-------------------------|
| B | Total boron uptake | 426.8 mg |
| | Total boron in wood | 58.81 mg |
| | Boron mass fraction in wood | 0.138 |
| | Total boron in leachate | 367.5 mg |
| | Boron mass fraction in leachate | 0.862 |
| | Mass balance | 99.88 % |
| | Boron retention after leaching | 0.464 kg/m ³ |
| Zn | Total zinc uptake | 1645 mg |
| | Total zinc in wood | 1476 mg |
| | Zinc mass fraction in wood | 0.929 |
| | Total zinc in leachate | 112.0 mg |
| | Zinc mass fraction in leachate | 0.071 |
| | Mass balance | 96.57 % |
| | Zinc retention after leaching | 11.66 kg/m ³ |
| Cu | Total copper uptake | 4076 mg |
| | Total copper in wood | 4035 mg |
| | Copper mass fraction in wood | 0.955 |
| | Total copper in leachate | 189.9 mg |
| | Copper mass fraction in leachate | 0.045 |
| | Mass balance | 103.6 % |
| | Copper retention after leaching | 31.86 kg/m ³ |

CLAIMS:

1. A preserving composition which includes a basic zinc compound and a compound of boron.
5
2. A preserving composition according to claim 1 wherein the composition includes zinc ions and borate ions in an aqueous solution of ammonia.
10 3. A preserving composition according to claim 1 or claim 2 wherein the composition includes ions of copper.
4. A preserving composition according to claim 3 wherein the ions of copper are cupric ions.
15 5. A preserving composition according to claim 4 wherein the cupric ions are derived from copper sulfate.
6. A preserving composition according to any one of the preceding claims wherein the composition includes fluoride ions.
20
7. A preserving composition according to claim 6 wherein the fluoride ions are derived from potassium fluoride.
25 8. A preserving composition according to any one of the preceding claims wherein the composition includes a quaternary ammonium compound.
30 9. A preserving composition according to any one of the preceding claims wherein the composition comprises an aqueous solution containing from 5gms per litre to

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saturation of borate ions and from 10gms per litre to saturation of zinc ions.

10. A method of preserving a cellulosic compound by
5 impregnating the cellulosic compound with a preserving composition according to any one of claims 1 to 9.

11. A method according to claim 10 wherein the cellulosic compound is flooded with the preserving composition under
10 vacuum.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU01/00325

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. ⁷: B27K 3/32, 3/22, 3/16

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC⁷ AS ABOVE

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Derwent WPAT: IPC⁷ as above and boron+ and borat+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|---|-------------------------|
| X, Y | EP 450568 B1 (Kabushiki Kaisha Koshii Preserving) 2 August 1995 Whole document | 1 to 5, 8 to 11 6, 7 |
| X, Y | EP 636461 A1 (Kabushiki Kaisha Koshii Preserving) 1 February 1995 Whole document | 1 to 5, 8 to 11 6, 7 |
| X, Y | WO 9218007 A1 (Saneish Pty. Ltd.) 29 October 1992 Whole Document | 1 6, 7 |

Further documents are listed in the continuation of Box C See patent family annex

* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
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Date of the actual completion of the international search

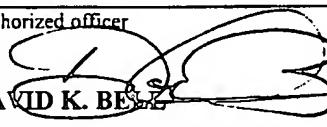
28 May 2001

Date of mailing of the international search report

28 MAY 2001

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/AU01/00325

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent Document Cited in Search Report | | | | Patent Family Member | | | |
|---|---------|----|----------|----------------------|----------|----|---------|
| WO | 9218007 | AU | 15680/92 | AU | 14840/92 | US | 5612046 |
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| EP | 636461 | AU | 68756/94 | CA | 2128983 | JP | 7088808 |
| | | NZ | 264079 | US | 5478598 | | |
| END OF ANNEX | | | | | | | |